



CASE PI/5-30958A

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF

Stephan Gutmann

APPLICATION NO: 10/039,706

FILED: November 9, 2001

FOR: Solvates of Pymetrozine

Art Unit: 1624

Examiner: Venkataraman Balasubramanian

RECEIVED

MAR 03 2004

Assistant Commissioner for Patents  
Washington, D.C. 20231

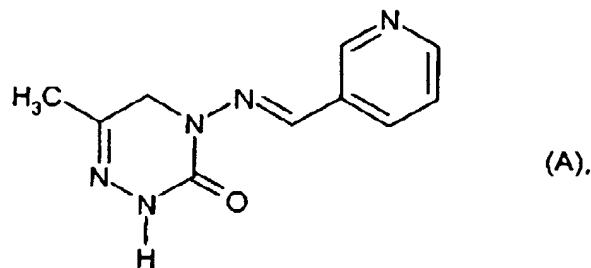
DECLARATION UNDER 37 CFR § 1.132

I, Thomas Rapold, a citizen of the Federal Republic of Germany and resident of Wallbach in the canton of Aargau, Switzerland, declare that:

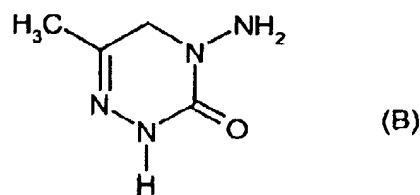
- 1) I was awarded the degree of a Master of Science in Organic Chemistry by the 'Universität des Saarlandes', Saarbrücken (Germany) in 1984, and the degree of a Doctor of Natural Science by the same University in 1987. I am presently employed with the company Syngenta Münchwilen AG, Münchwilen, Switzerland. I have been engaged in the screening and optimizing of organic syntheses for active ingredients and their intermediates by Syngenta Münchwilen AG (former Novartis AG) since 1987. I am well acquainted with the techniques and methods of optimizing reaction conditions and process parameters in order to develop new chemical procedures for pesticides. I now disclose the results of chemical experiments which were carried out in connection with developing the technology disclosed in U.S. patent 5,384,403.
- 2) I have been asked by the inventor of the invention described in the above-identified patent application to carry out the tests described below, and I have read and am familiar with the subject matter of the present application.
- 3) The following investigations were carried out under my supervision in a laboratory in Münchwilen, Switzerland, in order to optimize a chemical procedure for the production of the compound of the formula (A) below, the compound of Example 11 from the U.S. patent 5,384,403.

4) Test Procedure:

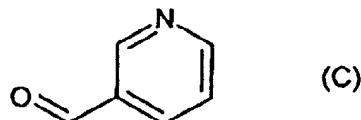
Preparation of the compound of the formula



330.1 g of a mixture containing 11.3 g % per weight (38.4 g, 0.300 moles) of the aminotriazinone of formula



in methanol was filled into a reactor. The mixture was heated up to 35°C and 173.5 g of the solvent distilled off at a pressure of between 120 and 190 mbar. The residue was cooled to 25°C and 107.1 g of a mixture containing 30.9 % per weight (33.1 g, 0.309 moles) of 3-pyridinealdehyde of the formula



in water were added over a time of 30 minutes. The mixture was stirred for a further 3.5 hours and the temperature kept at 25°C. A white suspension formed during that time. Then, the mixture was cooled to 5°C, stirred for 10 minutes, filtered and the filter cake washed with 100g water. The so obtained product was dried at 70°C.

The yield was 64.2 g of the crude compound of the formula (A).

5) Analysis of the compound of the formula (A):

Analytical method:

The contents of active ingredient of the formula (A) of the sample of the above example was determined in the following way:

Apparatus: Liquid chromatograph Spectra Physics SP 8800

Column: Nucleosil C18, length 250 mm

Column temperature: Room temperature

Injection volume: 10 $\mu$ l

Flow rate: 1 ml per minute

Mobile phase: Acetonitrile/water/phosphate buffer pH 7 (10 + 80 + 10)

Phosphate buffer: 0.041 mol/l Na<sub>2</sub>HPO<sub>4</sub> + 0.028 mol/l KH<sub>2</sub>PO<sub>4</sub> in water

Reference sample: Waterfree compound of the formula (A) with a known content

6) Results:

Content of compound of the formula (A):	97,7 %
Content of compound of formula (B)	< 0,1 %
Content of compound of formula (C)	< 0,1 %

7) It is my opinion that the compound of the formula (I) according to the present application, which is the compound of the above formula (A) containing in addition two molecules of water, could not have been expected from the teachings of US Patent No.5,384,403.

8) I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this patent application and any patent issuing thereon.

Signed this 24 day of February, 2004.

  
Thomas Rapold